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(54) Curable compositions and cured gel parts thereof

(57) A linear polyfluoro compound having at least two alkenyl groups as a base component is blended with a linear polyfluoro compound having one alkenyl group and a hydrosilyl-bearing linear polysiloxane having fluoroalkyl substituents in side chains and at ends thereof. With a platinum catalyst added, there is obtained a curable composition having improved storage stability which will quickly cure into satisfactory gel products.

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Description

[0001] This invention relates to curable fluoro compositions, particularly with a view to achieving storage stability and curability and more particularly, to such compositions as will cure to a gel.

BACKGROUND

[0002] Cured gel parts of silicone rubber have good electrical and thermal insulation, stable electrical properties, and softness. They find use as potting and sealing agents for electric and electronic parts and as coating materials on control circuit elements such as power transistors, IC's and capacitors for protecting the elements from external thermal and mechanical disturbances.

[0003] Typical of the silicone rubber compositions forming such cured gel parts are organopolysiloxane compositions of the addition curing type. For example, JP-A 143241/1981, 3959/1987, 35655/1988 and 33475/1988 corresponding to USP 4,771,119 disclose compositions comprising an organopolysiloxane having a vinyl group attached to a silicon atom and an organohydrogenpolysiloxane having a hydrogen atom attached to a silicon atom wherein the compositions undergo crosslinking reaction in the presence of platinum catalysts to form silicone gel.

[0004] The silicone gel products resulting from these organopolysiloxane compositions, however, are susceptible to such chemicals as strong bases and strong acids and such solvents as toluene, alcohol and gasoline and cannot sustain their performance on account of swelling or degradation.

[0005] To solve this problem, a fluorochemical gel composition comprising a polyfluoro compound based on a divalent perfluoroalkylene or perfluorooxyalkylene group and having two alkenyl groups in a molecule as a base component, an organohydrogenpolysiloxane having a hydrogen atom attached to a silicon atom, and a platinum group catalyst was proposed as well as a cured fluorochemical gel part obtained by curing the composition. This fluorochemical gel shows improved chemical resistance and solvent resistance over silicone gel as intended. However, the composition lacks long-term storage stability or is short in the steady curing of quickly reaching the desired penetration.

[0006] The general aim herein is to provide new and useful curable fluoro/silicone compositions, cured products made from them, and methods of making them. A preferred object is to provide curable compositions having good storage stability and sufficient curability to ensure that they quickly cure to a cured gel product having a suitable penetrability. Another object of the invention is to provide a cured gel product obtained by curing the fluorochemical curable composition.

[0007] We have found that by using a linear polyfluoro compound having at least two alkenyl groups as a base component, and combining it with a linear polyfluoro compound having one alkenyl group and a hydrosilyl-bearing linear polysiloxane having fluoroalkyl substituents in side chains and at ends thereof, we can obtain a curable composition having good or improved storage stability and good or improved curability or curing stability, and which will cure into satisfactory gel parts.

[0008] Specifically, the present invention provides a fluorochemical curable composition comprising as main components,

(A) linear polyfluoro compound of the following general formula (1),
(B) linear polyfluoro compound of the following general formula (2),
(C) hydrosilyl-bearing organic silicon compound of the following general formula (3), and
(D) a catalytic amount of platinum group catalyst.

[0009] The invention also provides a cured gel part obtained by curing the composition, preferably having a penetration of 1 to 200 according to ASTM D-1403.

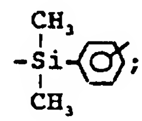
$$CH_2=CH-(X)_a-Rf^1-(X)_a-CH=CH_2 \quad (1)$$

$$Rf^2-(X)_a-CH=CH_2 \quad (2)$$

$$Rf^3-(X)_a-CH_2CH_2\underset{CH_3}{\overset{CH_3}{\underset{|}{\overset{|}{Si}}}}O-\left(\underset{CH_3}{\overset{H}{\underset{|}{\overset{|}{Si}}}}O\right)_k-\left(\underset{CH_3}{\overset{CH_3}{\underset{|}{\overset{|}{Si}}}}O\right)_m-\left(\underset{CH_3}{\overset{CH_2-(X)_a-Rf^4}{\underset{|}{\overset{|}{Si}}}}O\right)_n-\underset{CH_3}{\overset{CH_3}{\underset{|}{\overset{|}{Si}}}}-CH_2CH_2-(X)_a-Rf^3 \qquad (3)$$

[0010] In the formulas, X is independently $-CH_2-$, $-CH_2O-$, $-CH_2OCH_2-$ or $-Y-NR^1-CO-$;

Y is $-CH_2-$ or



R^1 is hydrogen or substituted or unsubstituted monovalent hydrocarbon group;
Rf^1 is a divalent perfluoroalkylene or perfluorooxyalkylene group; Rf^2 is a monovalent perfluoroalkyl or perfluorooxyalkyl group; Rf^3 and Rf^4 are independently as defined for Rf^2;
letter a is independently equal to 0 or 1, k is an integer of at least 2, m is an integer of at least 1, and n is an integer of at least 1.

BRIEF DESCRIPTION OF THE DRAWINGS

[0011]

The only figure, FIG. 1 is a graph showing the curing behavior at 150°C of the curable compositions of Example 1 and Comparative Examples 1 and 2.

DETAILED DESCRIPTION

[0012] Component (A) of the curable composition according to the invention is a linear polyfluoro compound of the following general formula (1).

$$CH_2=CH-(X)_aRf^1-(X)_a-CH=CH_2 \qquad (1)$$

[0013] In formula (1), Rf^1 is a divalent perfluoralkylene or divalent perfluorooxyalkylene group. Preferred divalent perfluoroalkylene groups are represented by
$-C_mF_{2m}-$ wherein m is an integer of 1 to 10, preferably 2 to 6. Preferred divalent perfluorooxyalkylene (or perfluoropolyether) groups are represented by the following formulas:

$$-(\underset{Y}{\underset{|}{C}}FOCF_2)_p-(CF_2)_r-(CF_2OC\underset{Y}{\underset{|}{F}})_q$$

wherein Y is fluorine or a CF_3 group, letters p, q and r are integers in the ranges: $p \geq 1$, $q \geq 1$, $2 \leq p+q \leq 200$, and $0 \leq r \leq 6$, and especially $2 \leq p+q \leq 110$,

$$-CF_2CF_2OCF_2-(\underset{CF_3}{\underset{|}{C}}FOCF_2)_s-(CF_2)_r-(CF_2O\underset{CF_3}{\underset{|}{C}}F)_t-CF_2OCF_2CF_2-$$

wherein r, s, and t are integers in the ranges: $0 \le r \le 6$, $s \ge 0$, $t \ge 0$, and $0 \le s+t \le 200$, and especially $2 \le s+t \le 110$;

$$-\underset{Y}{\underset{|}{C}}F-(O\underset{Y}{\underset{|}{C}}FCF_2)_u-(OCF_2)_v-O\underset{Y}{\underset{|}{C}}F-$$

wherein Y is fluorine or a CF_3 group, and u and v are integers in the ranges: $1 \le u \le 100$ and $1 \le v \le 50$; and

$-CF_2CF_2-(OCF_2CF_2CF_2)_w-OCF_2CF_2-$ wherein w is an integer in the range: $1 \le w \le 100$.

[0014] Illustrative examples of Rf^1 include the following groups:

$-C_4F_8-$, $-C_6F_{12}-$,

$$-(\underset{CF_3}{\underset{|}{C}}FOCF_2)_n(CF_2O\underset{CF_3}{\underset{|}{C}}F)_m-$$

$\overline{n+m} = 2 \sim 200$, $-CF_2CF_2OCF_2(CF_2)_2CF_2OCF_2CF_2-$,

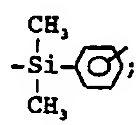
$$-CF_2CF_2OCF_2\underset{CF_3}{\underset{|}{C}}FOCF_2(CF_2)_2CF_2O\underset{CF_3}{\underset{|}{C}}FCF_2OCF_2CF_2- ,$$

$-CF_2(OCF_2CF_2)_n(OCF_2)_mOCF_2-$ $\overline{n} = 1 \sim 100$, $\overline{m} = 1 \sim 100$,

$$-\underset{CF_3}{\underset{|}{C}}F(O\underset{CF_3}{\underset{|}{C}}FCF_2)_n(OCF_2)_mO\underset{CF_3}{\underset{|}{C}}F-$$

$\overline{n} = 1 \sim 100$, $\overline{m} = 1 \sim 100$, $-CF_2CF_2(OCF_2CF_2CF_2)_nOCF_2CF_2-$ $\overline{n} = 5 \sim 100$

[0015] In formula (1), X is independently $-CH_2-$, $-CH_2O-$, $-CH_2OCH_2-$ or $-Y-NR^1-CO-$. Herein, Y is $-CH_2-$ or a group represented by



;

[0016] R^1 is hydrogen or substituted or unsubstituted monovalent hydrocarbon group. Preferred monovalent hydrocarbon groups are those having 1 to 12 carbons, and more preferably 1 to 10 carbons, for example, alkyl groups such as methyl, ethyl, propyl, butyl, hexyl, cyclohexyl, and octyl; aryl groups such as phenyl and tolyl; and aralkyl groups such as benzyl and phenylethyl. Also included are substituted hydrocarbon groups in which some or all of the hydrogen atoms of the foregoing groups are replaced with halogen atoms such as fluorine.

[0017] The alkenyl groups that formula (1) possesses are preferably those groups having a $-CH=CH_2$ structure at an end, for example, vinyl and allyl groups. The alkenyl groups may be attached to the backbone at both ends directly or via a divalent linking group represented by X.

[0018] In formula (1), letter a is independently equal to 0 or 1.

[0019] Component (B) of the composition according to the invention is a linear polyfluoro compound or clip following general formula (2).

$$Rf^2\text{-}(X)_a\text{-}CH{=}CH_2 \qquad (2)$$

[0020] Herein Rf^2 is a monovalent perfluoroalkyl group or a monovalent perfluorooxyalkyl (or perfluoropolyether) group. Preferred monovalent perfluoroalkyl groups are represented by C_mF_{2m+1}- wherein m is an integer from 1 to 20, and preferably from 2 to 10. Preferred illustrative examples of monovalent perfluorooxyalkyl groups are given below.

$$F\text{-}(\underset{\displaystyle CF_3}{\underset{|}{C}}FCF_2O)_p\text{-}\underset{\displaystyle CF_3}{\underset{|}{C}}F\text{-}$$

p is an integer of at least 1.

$$F\text{-}(\underset{\displaystyle CF_3}{\underset{|}{C}}FCF_2O)_q\text{-}\underset{\displaystyle CF_3}{\underset{|}{C}}FCF_2OCF_2CF_2\text{-}$$

q is an integer of at least 1.

[0021] Illustrative examples of Rf^2 are given below.

C_4F_9-, C_8F_{17}-

$$F\text{-}(\underset{\displaystyle CF_3}{\underset{|}{C}}FCF_2O)_n\text{-}\underset{\displaystyle CF_3}{\underset{|}{C}}F\text{-} \qquad n = 2\text{-}200$$

$$F\text{-}(\underset{\displaystyle CF_3}{\underset{|}{C}}FCF_2O)_n\text{-}\underset{\displaystyle CF_3}{\underset{|}{C}}FCF_2OCF_2CF_2\text{-} \qquad n = 2\text{-}200$$

[0022] In formula (2), X and "a" are as defined in formula (1). It is understood that X and "a" in formula (1) may be identical with or different from X and "a" in formula (2).

[0023] As in formula (1), the alkenyl groups that formula (2) possesses are preferably those groups having a $-CH{=}CH_2$ structure at an end, for example, vinyl and allyl groups. The alkenyl groups may be attached to the backbone directly or via a divalent linking group represented by X.

[0024] In order that the composition or the invention be used in casting, potting, coating, impregnation, adhesion or bonding, the composition should preferably have appropriate fluidity and appropriate physical properties when cured. It is desirable from this point of view that components (A) and (B) have a viscosity of about 5 to about 100,000 centipoise (cp) at 25°C. By selecting components (A) and (B), the composition is adjusted to the most appropriate viscosity within this range for a particular application.

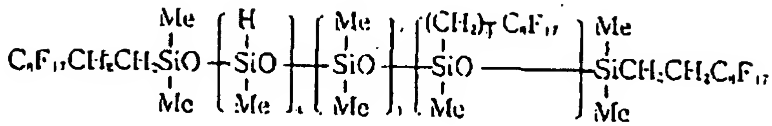
[0025] Component (C) of the composition according to the invention is a hydrosilyl-bearing organic silicon compound of the following general formula (3).

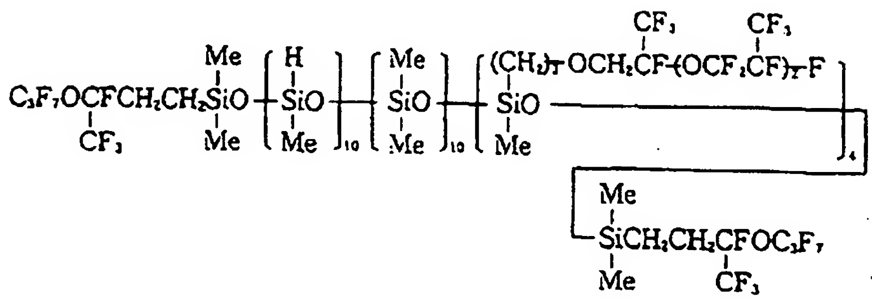
$$Rf^{3}-(X)_{a}-CH_{2}CH_{2}\underset{CH_{3}}{\overset{CH_{3}}{\underset{|}{\overset{|}{Si}}}}O-\left(\underset{CH_{3}}{\overset{H}{\underset{|}{\overset{|}{Si}}}}O\right)_{k}-\left(\underset{CH_{3}}{\overset{CH_{3}}{\underset{|}{\overset{|}{Si}}}}O\right)_{m}-\left(\underset{CH_{3}}{\overset{\overset{CH_{2}-(X)_{a}-Rf^{4}}{|}}{\overset{CH_{2}}{\underset{|}{\overset{|}{Si}}}}}O\right)_{n}-\underset{CH_{3}}{\overset{CH_{3}}{\underset{|}{\overset{|}{Si}}}}-CH_{2}CH_{2}-(X)_{a}-Rf^{3} \quad (3)$$

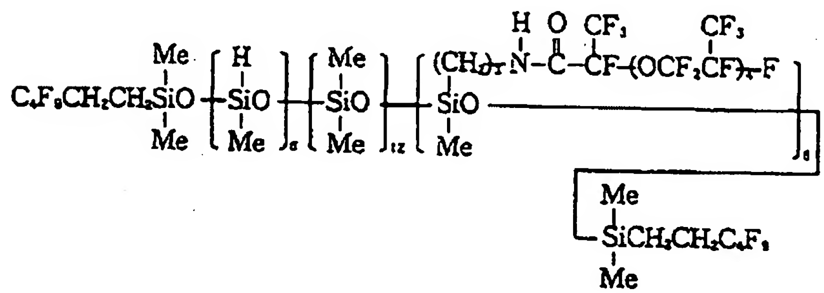
[0026] Herein, X and "a" are as defined above. More particularly, a plurality of X's and a plurality of "a's" in formula (3) may be identical or different from each other, and may also be identical with or different from X's and "a's" in formulas (1) and (2). Rf^3 and Rf^4 are independently as defined for Rf^2. Rf^3 may be identical with or different from Rf^4.

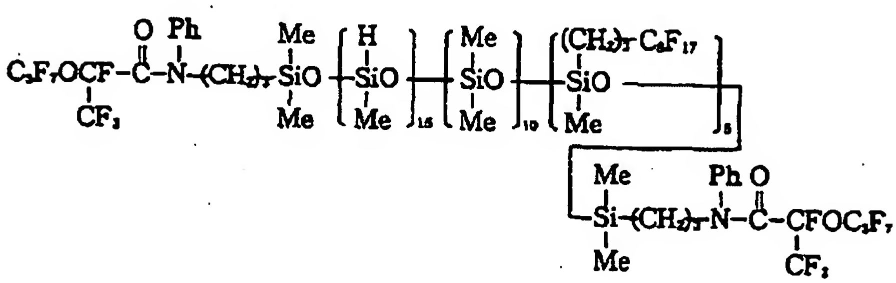
[0027] In formula (3), letter k is an integer of at least 2, preferably 2 to 20; m is an integer of at least 1, preferably 1 to 40; and n is an integer of at least 1, preferably 1 to 20.

[0028] Illustrative examples of the fluorinated organosiloxane are given below wherein Me is methyl and Ph is phenyl. These compounds may be used alone or in admixture.









[0029] We find that one can thereby achieve long-term storage stability and the steady curing of quickly reaching the desired penetration, which are attributable to the structure of the hydrosilyl-bearing linear organosilicon compound having fluoroalkyl groups substituted in side chains and at ends as component (C).

[0030] First, the substitution of fluoroalkyl groups in side chains and at ends is effective for increasing the fluorine content in the molecule, thereby improving the compatibility with components (A) and (B). Advantages derived from the improved compatibility are that the composition can be kept stable without, separation, and constant reaction proceeds during curing, resulting in a gel-like composition of quality.

[0031] Secondly, since hydrosilyl groups are positioned solely in side chains of the linear polysiloxane molecule, addition reaction for curing is moderate and a composition obtained by mixing components (A) to (D) can be kept stable. In particular, when a control agent for the hydrosilylation catalyst to be described later is blended in the composition, long-term storage stability is ensured. In contrast, a polysiloxane containing a hydrosilyl group at an end of the molecule as shown by $-OSi(CH_3)_2H$ has too high addition reactivity, allowing a composition obtained by mixing components (A) to

(D) to cure sooner or later to a gel state even when a control agent is admixed. In this case, it is unavoidable to formulate the composition in two-part form, for example, by blending components (A), (B) and (D) as one part, and separately blending components (A), (B) and (C) as another part so that two parts are mixed on use. In this regard, the invention permits all the components to be formulated as a one-part composition, avoiding a cumbersome step of mixing before use.

[0032] Thirdly, hydrosilyl-bearing siloxane units $-OSi(CH_3)H-$ are randomly distributed in the molecule along with dimethylsiloxane units $-OSi(CH_3)_2-$ and fluoroalkylsubstituted siloxane units, which is a further advantage. The random distribution allows the consumption of hydrosilyl groups during curing reaction to proceed smoothly, ensuring the steady curing of quickly reaching the desired penetration. In contrast, a linear polysiloxane of the structure in which only hydrosilyl-bearing siloxane units $-Osi(CH_3)H-$ are continuously repeated tends to prevent the reaction of all hydrosilyl groups from proceeding to completion, resulting in unsteady curing.

[0033] Preferably, component (C) is blended in such amounts as to provide 0.2 to 2.0 mol, more preferably 0.5 to 1.3 mol of hydrosilyl groups, that is, SiH groups, in component (C) per mol or the aliphatic unsaturated groups including vinyl, allyl and cycloalkenyl groups in the entire composition. Outside the range, less amounts of SiH groups would provide an insufficient degree or crosslinking and fail to cure into gel whereas excessive amounts of SiH groups can invite the risk of expansion during curing.

[0034] Component (D) of the composition according to the invention is a platinum group catalyst which promotes addition reaction between alkenyl groups in components (A) and (B) and hydrosilyl groups in component (C). The platinum group catalysts are generally noble metal compounds which are expensive, and relatively easily available platinum compounds are often used.

[0035] Illustrative examples of the platinum compound include chloroplatinic acid, complexes of chloroplatinic acid with olefins such as ethylene, complexes of chloroplatinic acid with alcohols or vinylsiloxanes, and platinum on silica, alumina or carbon. The platinum compound is not restricted to these examples. Known platinum group compounds other than the platinum compounds include compounds of rhodium, ruthenium, iridium and palladium, for example, $RhCl(PPh_3)_3$, $RhCl(CO)(PPh_3)_2$, $Ru_3(CO)_{12}$, $IrCl(CO)(PPh_3)_2$, and $Pd(PPh_3)_4$. The catalyst is usually employed in an amount of 0.1 to 100 ppm based on the total weight of components (A), (B) and (C).

[0036] In the composition of the invention comprising components (A) to (D) mentioned above, various e.g. per se known additives may be added. Such additives include control agents for hydrosilylation catalysts, for example, acetylene alcohols such as 1-ethynyl-1-hydroxycyclohexane, 3-methyl-1-butyn-3-ol, 3,5-dimethyl-1-hexyn-3-ol, 3-methyl-1-pentyn-3-ol, and phenyl butynol; 3-methyl-3-penten-1-yne, 3,5-dimethyl-3-hexen-1-yne, polymethylvinylsiloxane cyclic compounds, and organic phosphorus compounds. These control agents are effective for maintaining adequate curing reactivity and storage stability. Further, the cured gel resulting from the composition of the invention can be adjusted in hardness and mechanical strength by adding thereto inorganic fillers such as fumed silica, silica aerosil, precipitated silica, ground silica diatomaceous earth, iron oxide, zinc oxide, titanium oxide, calcium carbonate, magnesium carbonate, zinc carbonate, and carbon black. Of course, hollow inorganic fillers, hollow organic fillers and spherical rubbery fillers may also be added. The amounts of these additives blended are arbitrary as long as the physical properties of cured gel are not adversely affected.

[0037] The composition of the invention comprising the essential components (A) to (D) and optional components as described above can be cured into a gel product having resistance to solvents and chemicals.

[0038] The term (cured) gel product indicates a state having in part a three-dimensional structure and exhibiting deformation and fluidity under applied stresses. As a general measure, the gel product should have a hardness of "0" or lower as measured by a JIS rubber hardness meter and a penetration of a loaded needle of 1 to 200 according to ASTM D-1403 (1/4 cone).

[0039] Conventional well-known methods may be used in forming a cured gel product from the composition of the addition curing type according to the invention. For example, the composition is introduced into a suitable mold where the composition is cured. Alternatively, the composition is applied onto a suitable substrate and then cured. Curing is readily effected by heating at a temperature of about 60°C to about 150°C for about 30 to about 180 minutes.

[0040] There has been described a curable composition which remains well stable during storage and is effectively curable. The resulting cured gel product can have improved properties.

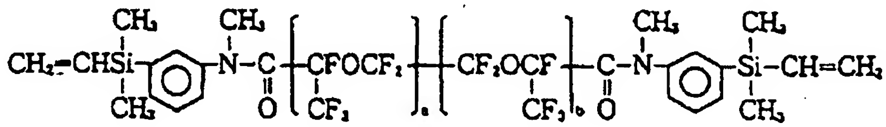
EXAMPLE

[0041] Examples of the invention are given below by way of limitation and not by way of limitation. All parts are by weight. Viscosity is a measurement in centipoise (cp) at 25°C.

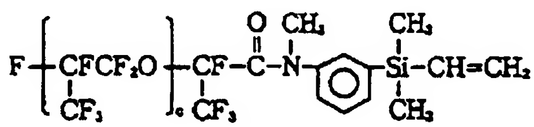
Example 1

[0042] To 50 parts or a polymer (viscosity 3,000 cp) of formula (4) shown below and 50 parts of a polymer (viscosity

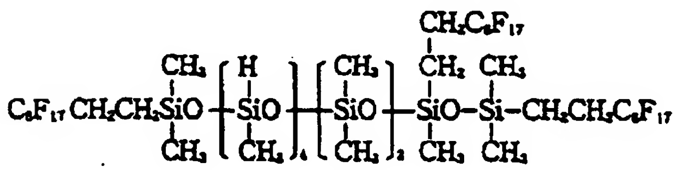
1,000 cp) or formula (5) shown below were added 13.7 parts of a compound of formula (6) shown below, 0.15 part of a 50% toluene solution of ethynyl cyclohexanol, and 0.015 part of an ethanol solution of a chloroplatinic acid/vinylsiloxane complex (platinum concentration 3.0% by weight). By mixing the ingredients, a fluorochemical composition was prepared.



$\overline{a+b} = 35$ ... (4)



$\overline{c} = 24$ ... (5)

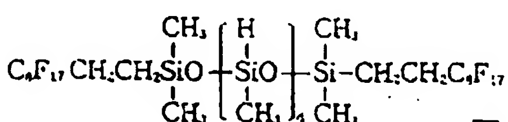


... (6)

[0043] The composition was heated at 150°C for one hour for curing into a clear gel product which showed a penetration of 78 as measured according to ASTM D-1403 (1/4 cone).

Comparative Example 1

[0044] A composition was prepared as in Example 1 except that 8.7 parts of a compound of formula (7) shown below was used instead of the compound of formula (6).



... (7)

$\overline{d} = 4$

[0045] The composition was similarly cured, obtaining a clear gel product having a penetration of 64.

Comparative Example 2

[0046] A composition was prepared as in Example 1 except that 12.1 parts of a compound of formula (8) shown below was used instead of the compound of formula (6).

$$\left[C_8F_{17}CH_2CH_2CH_2\overset{CH_3}{\underset{CH_3}{|}}SiO\right]\left[H\overset{CH_3}{\underset{CH_3}{|}}SiO\right]_2 SiCH_2CH_2-C_6F_{12}-CH_2CH_2Si\left[O\overset{CH_3}{\underset{CH_3}{|}}SiH\right]_2\left[O\overset{CH_3}{\underset{CH_3}{|}}SiCH_2CH_2CH_2C_8F_{17}\right] \quad \cdots (8)$$

[0047] The composition was similarly cured, obtaining a clear gel product having a penetration of 65.

[0048] The above-prepared compositions were examined for storage stability and curability.

<u>Storage stability</u>

[0049] The compositions were stored in sealed containers at 40°C for 30 days. The aged compositions were measured for viscosity, with the results shown in Table 1.

Table 1

	E1	CE1	CE2
Initial (cp)	1140	1290	1250
Aged (cp)	1140	1290	gelled

<u>Curability</u>

[0050] The compositions were heated at 150°C for curing while they were measured for modulus of elasticity (G') at predetermined intervals. The results are shown in FIG. 1.

[0051] It is evident that the composition of Comparative Example 1 does not undergo quick curing to completion and lacks steady curing. The composition of Comparative Example 2 lacks long-term storage stability. The composition within the scope of the invention satisfies both or storage stability and quick curing.

[0052] Japanese Patent Application No. 366499/1997 is incorporated herein by reference.

[0053] Reasonable modifications and variations are possible from the foregoing disclosure without departing from either the spirit or scope of the present invention.

Claims

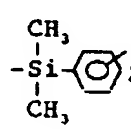
1. A curable composition comprising

(A) linear polyfluoro compound of the following general formula (1):

$$CH_2=CH-(X)_a-Rf^1-(X)_a-CH=CH_2 \quad (1)$$

wherein X is independently $-CH_2-$, $-CH_2O-$, $-CH_2OCH_2-$ or $-Y-NR^1-CO-$,

Y is $-CH_2-$ or



;

R^1 is hydrogen or substituted or unsubstituted monovalent hydrocarbon group,

Rf^1 is a divalent perfluoroalkylene or perfluorooxyalkylene group, and letter a is independently equal to 0 or 1,

(B) linear polyfluoro compound of the following general formula (2):

$$Rf^2\text{-}(X)_a\text{-}CH{=}CH_2 \qquad (2)$$

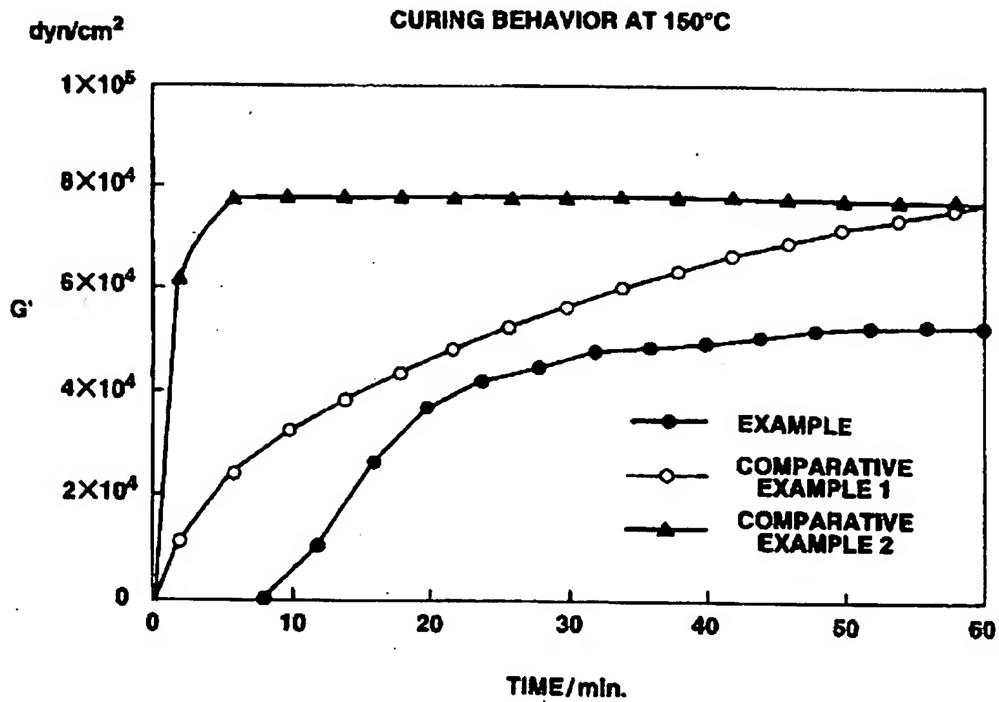
wherein X and "a" are as defined above, Rf^2 is a monovalent perfluoroalkyl or perfluorooxyalkyl group,
(C) hydrosilyl-bearing organic silicon compound of the following general formula (3):

$$Rf^3\text{-}(X)_a\text{-}CH_2CH_2\overset{\displaystyle CH_3}{\underset{\displaystyle CH_3}{Si}}O\left(\overset{\displaystyle H}{\underset{\displaystyle CH_3}{Si}}O\right)_k\left(\overset{\displaystyle CH_3}{\underset{\displaystyle CH_3}{Si}}O\right)_m\left(\overset{\displaystyle CH_2\text{-}(X)_a\text{-}Rf^4}{\overset{\displaystyle CH_2}{\underset{\displaystyle CH_3}{Si}}}O\right)_n\overset{\displaystyle CH_3}{\underset{\displaystyle CH_3}{Si}}\text{-}CH_2CH_2\text{-}(X)_a\text{-}Rf^3 \qquad (3)$$

wherein X and "a" are as defined above, Rf^3 and Rf^4 are independently as defined for Rf^2, letter k is an integer of at least 2, m is an integer of at least 1, and n is an integer of at least 1, and
(D) a catalytic amount of platinum group catalyst.

2. A gel product obtained by curing the composition or claim 1 and having a penetration or 1 to 200 according to ASTM D-1403.

FIG.1


CURING BEHAVIOR AT 150°C
dyn/cm2
1X10^5
8X10^4
6X10^4
4X10^4
2X10^4
0
G'
0
10
20
30
40
50
60
TIME/min.
EXAMPLE
COMPARATIVE EXAMPLE 1
COMPARATIVE EXAMPLE 2